

STRUCTURAL AND ELECTROMAGNETIC STUDY OF FERROMAGNETIC SEMICONDUCTOR MATERIAL

A thesis submitted to the partial fulfilment of the requirement
for the degree of

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IN

PHYSICS

BY

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CERTIFICATE

This is to certify that the dissertation entitled “**structural and electromagnetic study of ferromagnetic semiconductor material**” being submitted by **Mr .Bhabani Sankar Lenka** to the Department of Physics & Astronomy, National Institute of Technology, Rourkela, Orissa, for the award of the degree of Master of Science is a record of genuineresearch carried out by him under my supervision and guidance.

To the best of my knowledge, the matter embodied in the dissertation has not been submitted to any other University / Institute for the award of any Degree or Diploma.

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ABSTRACTS

A detail structural and electrical analysis of a few doubleperovskite (**La₂CoMnO₆**, **La₂FeMnO₆**) and their perovskite counterpart (**LaCoO₃**, **LaCrO₃**and **LaFeO₃**) have been performed. All the compounds are synthesized by wet chemical route(especially by sol-gel & pyrophoric route). From XRD data analysis, the samples are found to be in single phase and stoichiometric. The information about the molecular bonding is obtained from the FTIR spectra. The band gap is calculated from UV-Visible spectra and the sample is characterised whether it is semiconducting and insulating. The morphology of the materials has been studied through FESEM and finally the dielectric spectroscopy has been carried out for all compounds.

ACKNOWLEDGEMENT

On the submission of my thesis report titled “**structural and electromagnetic study of ferromagnetic semiconductor material**” I would like to convey my gratitude and sincere thanks to my supervisor Prof. Suryanarayan Dash, Department of Physics for his constant motivation and support during the course of my work in the last one year. I truly appreciate and value his esteemed guidance and encouragement from beginning to the end of this thesis. I am indebted to him for having helped me, shape the problem and providing insights towards the solution. I would also likely to acknowledge Mr Ranjit Kumar Panda for dielectric measurement.

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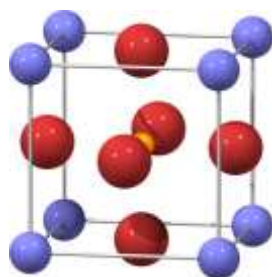
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1.1 Transitional metal oxides and their properties

Transition metal oxides belong to the class of material that contain transition element and oxygen. Insulators as well as metals are included under this category. Transition metal oxides have a useful application in a wide variety of technologically important catalytic processes. For example:- selective oxidation, reduction and dehydrogenation. A promising family of mixed transition metal oxides designated as $A_xB_{3-x}O_4$ ($A, B = \text{Co, Ni, Zn, Mn, Fe}$ etc.) play significant role for low cost and environmentally friendly energy storage & conservation technology. The transition metal oxides form one of the most interesting class of solid materials exhibiting a variety of structure and properties. The nature of the metal oxygen bonding can be varied from ionic to covalent to metallic. The unique nature of the outer d-electron generate the unusual properties in transition metal oxides. We find oxides with metallic properties ($\text{ReO}_3, \text{LaNiO}_3$) at one end of the range and oxides with highly insulating behaviour at the other end (BaTiO_3). Some oxides also transit both the regime with change in temperature, pressure, composition. Interesting electrical properties arise from charge ordering and defect ordering. Some oxides show divergence in magnetic properties ferromagnetism ($\text{CrO}_2, \text{LaSrMnO}_3$) – antiferromagnetism (LaCrO_3) transition.

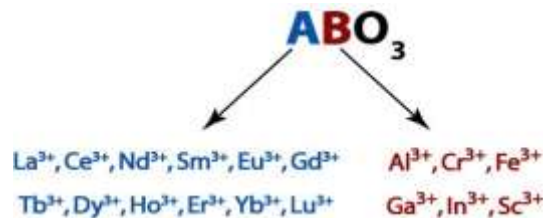
1.2 Perovskite structure

General chemical formula of perovskite are ABX_3 with A, B are two cations of unequal size and X is the anion. A is larger in size as compared to B. The ideal cubic-symmetry structure has the B cation in 6-fold coordination, surrounded by an octahedron of anions, and the A cation in 12-fold cuboctahedral coordination.



[Cubic perovskite unit cell. Blue spheres represent the A cations, yellow spheres represent the B cations, and red spheres represent oxygen anions forming an octahedra.]

The perovskite structure formed by many oxides that have the chemical formula ABO_3 . In the idealized cubic unit cell of such a compound, type 'A' atom sits at cube corner positions (0, 0, 0), type 'B' atom sits at body centre position (1/2, 1/2, 1/2) and oxygen atoms sit at face centred positions (1/2, 1/2, 0). The ions occupying the A,B lattice symmetry are shown below.



ABX_3 perovskite family has wide research attention due to its structural variation, which results in important physical properties which are useful in:-

- Superconductive & magneto resistive oxides
- Microwave dielectric materials
- Ferroelectrics
- Piezoelectric materials

1.3 Double Perovskites

To enhance electronic & magnetic properties, we can extend the perovskite family, particularly of A & B site cations. By substituting cation B' for B leads in general to solid $AB_{1-x}B'_xX_3$, but if $x \cong 0.5$ B & B' are sufficiently different in charge and size. Doubling of the ideal perovskite leads to another cubic structure with unit cell edge double that of ABO_3 perovskite. That's why these are called double perovskites. Self-ordered $A_2BB'O_6$ (e.g., A = La, B=Fe, Ni, Co, Cr and B' = Mn) double perovskites provide a unique alternative opportunity to control and/or to induce such behavior in oxides.

1.4 Antiferromagnetic Insulator

In antiferromagnetic materials, the magnetic moments of atoms or molecules related to the spins of electrons, align in a regular way with neighboring spins pointing in opposite directions. In this case the net magnetization is zero. Antiferromagnetism usually occurs at low temperature, Neel temperature above which the material shows paramagnetic nature. Antiferromagnetic solids show special behavior in an applied magnetic field.

depending upon the temperature. At very low temperatures, the solid exhibits no response to the external field, because the antiparallel ordering of atomic magnets is rigidly maintained. At higher temperatures, some atoms become free by breaking the orderly arrangement and align with the external field, so they behave generally as insulators. Antiferromagnetic materials are seen commonly among transition metal compounds, especially oxides. For examples- hematite, metals such as chromium, alloys such as iron manganese (FeMn), and oxides such as nickel oxide (NiO). There are also numerous examples among high nuclearity metal clusters. Organic molecules can also exhibit antiferromagnetic coupling under rare circumstances, as seen in radicals such as 5-dehydro-mxylylene.

1.5 Ferromagnetic semiconductor

Ferromagnetic materials show spontaneous magnetization. Magnetic moments are aligned in same direction without any external field. They have a finite magnetization. They are known as permanent magnets. Semiconductor materials have conductivity between metal & insulators. Their conductivity increases with increase in temperature. Its current conduction can be increased by doping these compound with impurities or dopants. An *N-type* semiconductor carries current mainly in the form of negatively-charged electrons, in a way similar to the current in a wire. A *P-type* semiconductor carries current predominantly as electron deficiencies called holes. A hole has a positive electric charge, equal and opposite to the charge on an electron. In a semiconductor material, the flow of holes occurs in a direction opposite to the flow of electrons.

Magnetic semiconductors are semiconductor materials that exhibit both ferromagnetism and useful semiconductor properties. When implemented in devices, these materials provide a new type of control of conduction. Traditional electronics are based on control of charge carriers (n- or p-type), practical magnetic semiconductor allows control of quantum spin state (up or down). This would theoretically provide near-total spin polarization, which is an important property for spintronic application.

1.6 Aim of the project work

In this report we studied the effect of Mn substitution in the B' site of the various double perovskite composition La_2BMnO_6 (B= Fe, Co, Cr etc.) through structural, electrical and rather morphological properties. In addition, we have compared with their perovskite counterpart LaBO_3 . The samples are prepared by wet chemical route especially by sol-gel and pyrophoric process. The structural characterization of the samples is carried out through detail Reitveld analysis of the XRD data which shows all the samples are in single phase and stoichiometric. The band gap is found out from UV-Visible spectroscopy different bonds in the compounds are detected from FTIR analysis. And their dielectric measurements are carried out to know their electrical properties.

2.1 Solid State Route

It is the most widely used method for the preparation of poly crystalline solids from a mixture of solid starting materials. Solids do not react together at room temperature over normal time scales and it is necessary to heat them to much higher temperatures, of the range 1000 to 1500 °C for a feasible reaction with an appreciable rate. Its an surface level phenomenon. By using this route we can synthesize compound upto micro level.

Steps-

- Selection of appropriate starting material which are reactive with fine grain powder and well defined composition.
- Weighing out the starting materials.
- Mixing the starting materials together by grinding with agate mortar or by ball milling.
- Pelletizing.
- Heating,grinding sample.
- Analyzing sample.
- If reaction is incomplete than from the step 3 it is repeated.

2.2 Wet Chemical Route

In wet chemical route starting materials are in liquid phase. It is the common name used for a group of synthesizing method used for synthesizing nano & ultra dispersed organic powders from aqueous and non aqueous solution. The group of methods are liquid phase sol gel process, hydrothermal synthesis, pechinimethod, spray drying, aerosol spray pyrolysis, cryochemical synthesis using liquid phase at one of the process stage. The main difference between solid state route & wet chemical route area:-

- Much smaller grains.
- Lower temperature.
- Shorter duration of phase transition.

Steps-

- Selection of appropriate starting materials.
- Mixing starting materials with suitable solvent (water ,nitric acid).
- Heating of the mixed solution till a dried viscous mass is formed.
- Grinding of the starting material to get homogeneous powder.
- Calcinations.
- Pelletizing.
- Sintering .
- Analyzing

2.2.1 Sol gel process:-

It is a wet chemical process for producing solid materials from small molecules. It is used for fabrication of metal oxides. Basic principle of this process involves the preparation of sol, which is a dispersion of solid particles, in liquid medium. The sol is prepared by mixing concentrated solution containing the cations of interest, with an organic solvent as dispersion medium. The sol is then destabilized by adding water. The presence of water modifies the pH of sol and reduces the repulsion between particles. This results in a large increase in viscosity of the medium, leading to the formation of gel. The next stage is removal of the dispersion medium. Again the rate at which the reaction is performed is important. Fast removal leads to a powder, this can be performed by passing fine droplets of gel through a column of alcohol. The final ceramic can be obtained by compaction & sintering of powderxerogel at an appropriate temperature.

The salient feature of sol-gel technique are given in five steps:-

- Stoichiometric proportion of high purity starting materials: rare earth oxides are taken and converted into their nitrates by using nitric acids. Later both the nitrates are dissolved in water to obtain clear solution. The resulting solution is heated to evaporate nitric acid.
- The second step is that conversion of all metal ions present in the solution as metal citrates by adding citric acid. Before conversion, one has to ensure that entire nitric acid present in the compound is completely evaporated. The method involves addition one mole of citric acid to each mole of metal ion present in the solution with constant stirring, so that all the metal ions completely converted into citrates.

Then ammonium hydroxide is added slowly to the solution by placing it in a cool water bath to avoid heat generation & this process is continued until the pH of the solution mixture reaches the value between 6-7, which is an important factor for gelation process. Then the solution is heated for evaporation for several hours until it becomes viscous mass.

- The third step of gelation process is gelation. For this purpose, ethylene glycol is added as gelating reagent. The gelating reagent is added to the viscous mass in the ratio of (metal to ethyl glycol) 1:1:2 & is heated on a hot plate for gelation, which converted into a grey coloured gel. The temperature required for gelation is between 60-200⁰c.
- The 5th step (final step) consists of pelletization & sintering process. The black coloured powder obtained after calcinations was pressed into pellets in cylindrical form with 1 cm. diameter and 2-4 mm thick ness applying pressure 5-7 tons/ sq. inch. During pelletization an organic material i.e. PVA has been used as binder.

2.2.2Pyrophoric process

It is a popular wet chemical method for fabrication of materials, favorably metal oxides. In this method aqueous solution of required amount of material are taken in stoichiometric proportion. The individual solution is heated up and concentric HNO₃ is added drop wise until it become clear. Then triethanolamine (TEA) with these solution in such ay that metal ion to TEA ratio is maintained at 1:1:2 to make viscous solution. The clear solution of TEA complexed metal nitrates was evaporated around 200⁰c with constant stirring. The constant heating of the solutions causing puffing & foaming. During evaporation the nitrates ions provides anoxidization environment for TEA which partially converts the hydroxyl group of TEA into carboxylic acid. When complete dehydration occur the nitrates themselves are decomposed with the evaporation of brown fumes of NO₂ leaving behind voluminous organic based black floppy powder i.e. precursor powder. The precursor powder after proper grinding are calcined at various temperature to get desired compound with variable particle size.

Steps:-

- Individual amount of constituent materials are taken in stoichiometric proportion.
- Individual solution is made by adding distilled water .

- These two solutions are mixed and completely stirred.
- TEA is added and heated at 60-200⁰c
- Black floppy mass is seen after ignition.
- The powder is grinded.
- Calcined.
- Pellets are made.
- sintered
- Analyzation of the sample.

2.3Calcination:-

The process of heating a substance to high temperature but below the melting point or fusing point for the purpose of some change in physical & chemical composition such as:-

- To drive off the water present as absorbed moisture, as water of crystallization.
- To drive off the carbon dioxide & other volatile constituents.
- To oxidize a part or whole of the substance

For calcinations three general class of furnace are used:- muffle, reverberatory, shaft furnace.

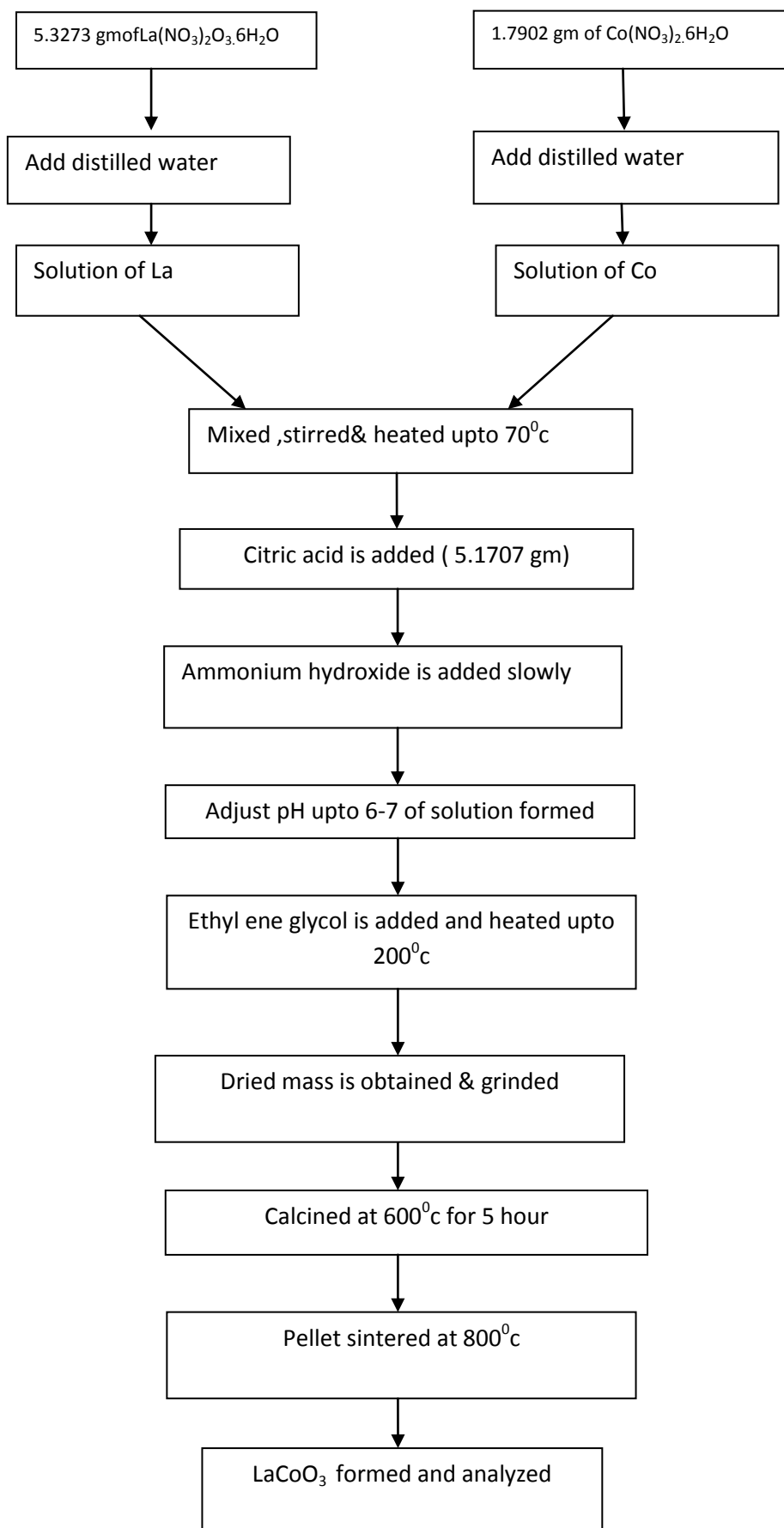
2.6 Sintering:-

It is a method of making object from powder substance, by heating material below its melting point until its particle adhere to each other. It is mostly used in ceramic objects, powder metallurgy, metal, plastic. Due to sintering we have the following advantages

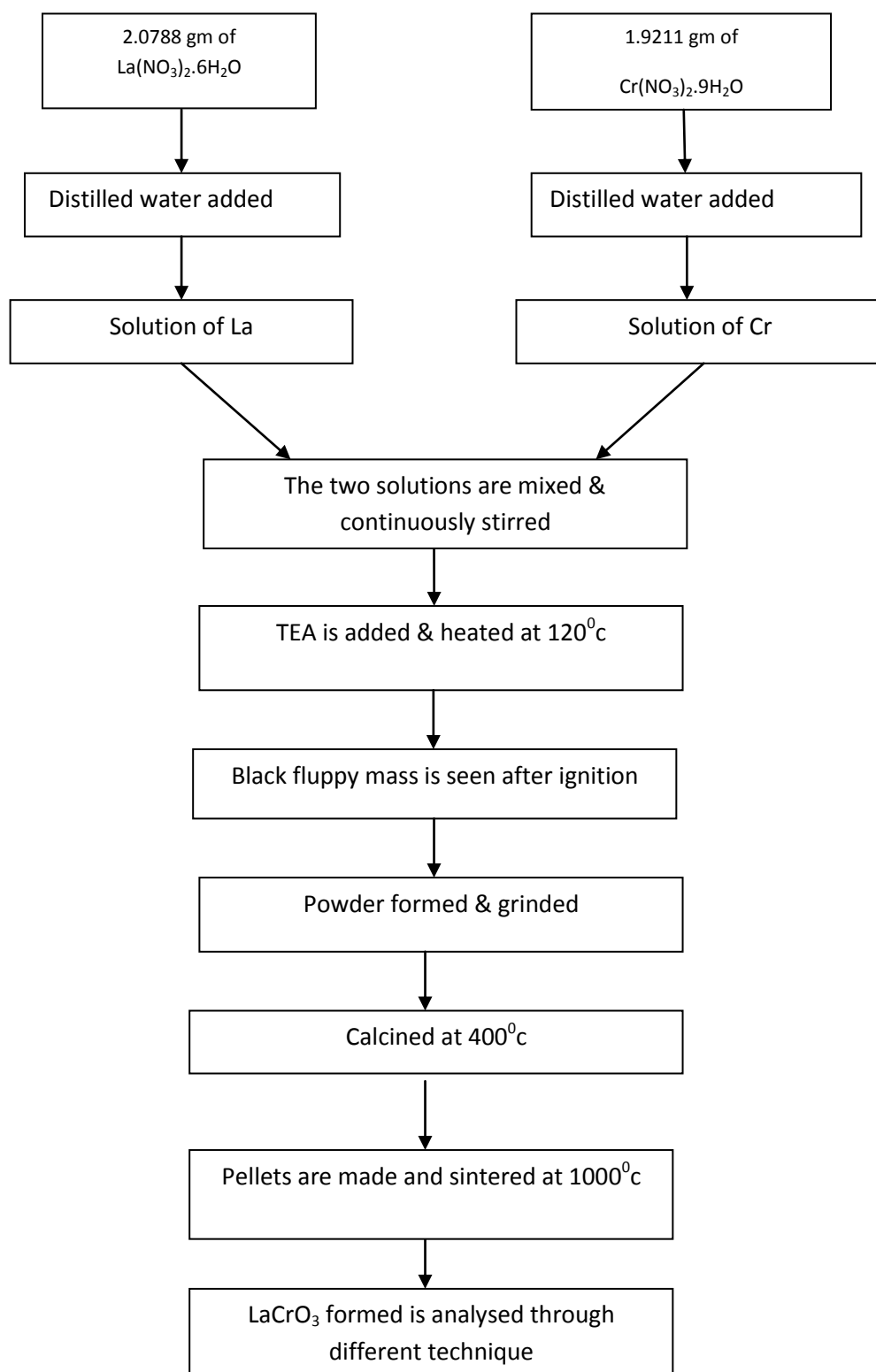
- High level purity & uniformity in starting material.
- Controlled grain size & directional elongation.
- Capability to produce material of controlled, uniform porosity and high strength.

2.7 preparations of LaCoO_3 , LaCrO_3 , LaFeO_3 , $\text{La}_2\text{CoMnO}_6$, $\text{La}_2\text{FeMnO}_6$

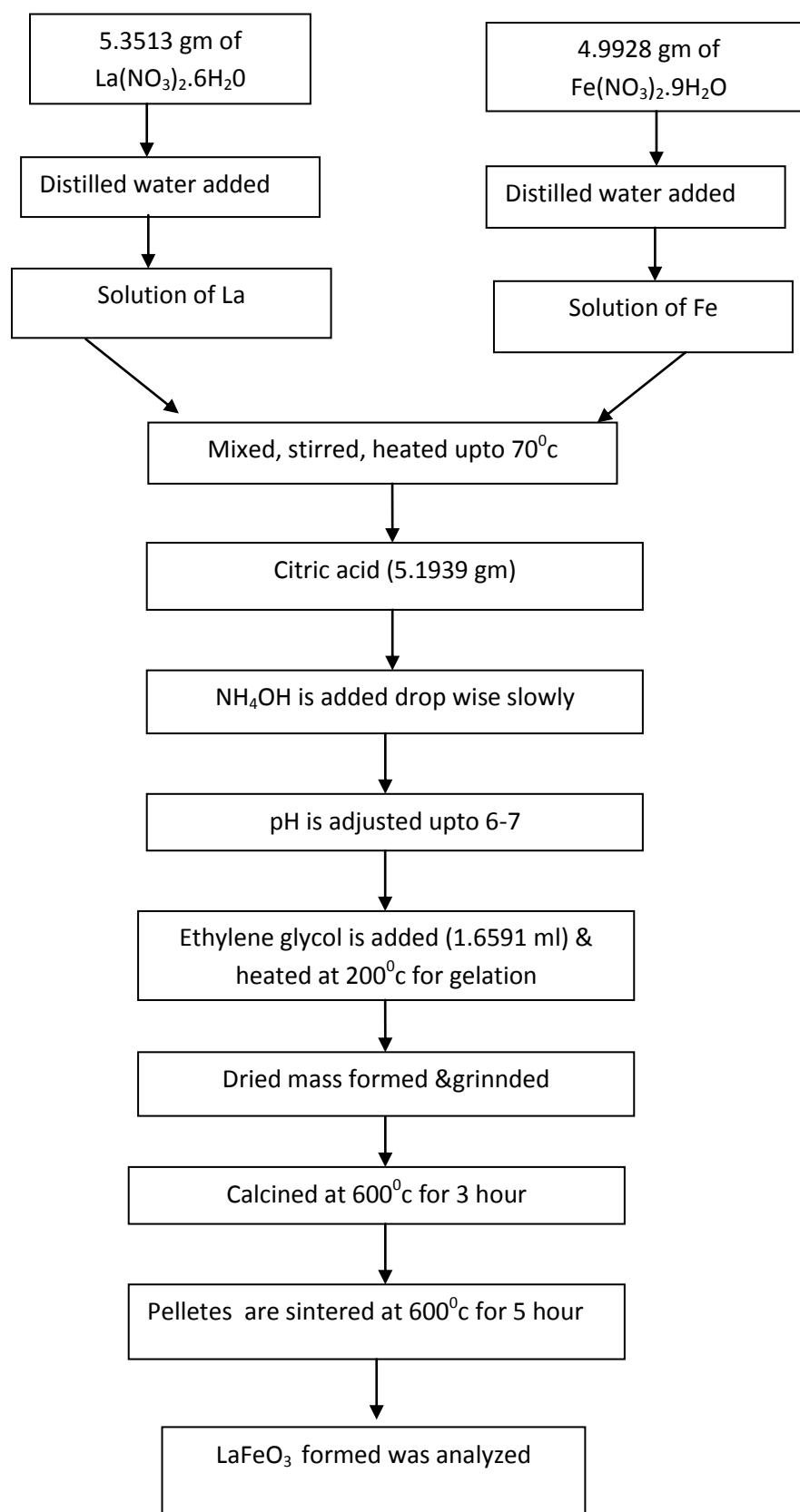
2.7.1 Synthesis of LaCoO_3 by sol-gel route:-



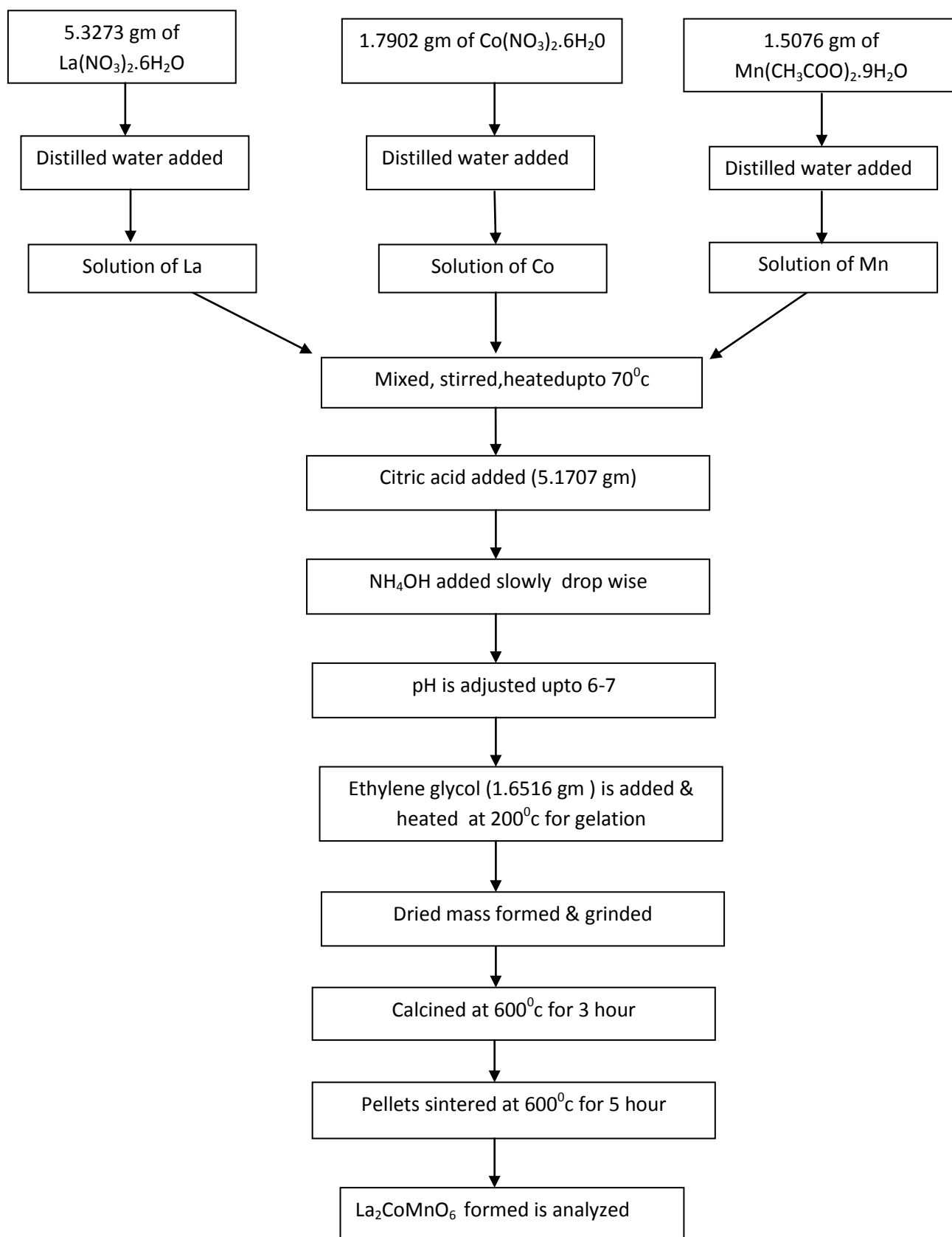
2.7.2 Synthesis of LaCrO_3 by pyrophoric process:-



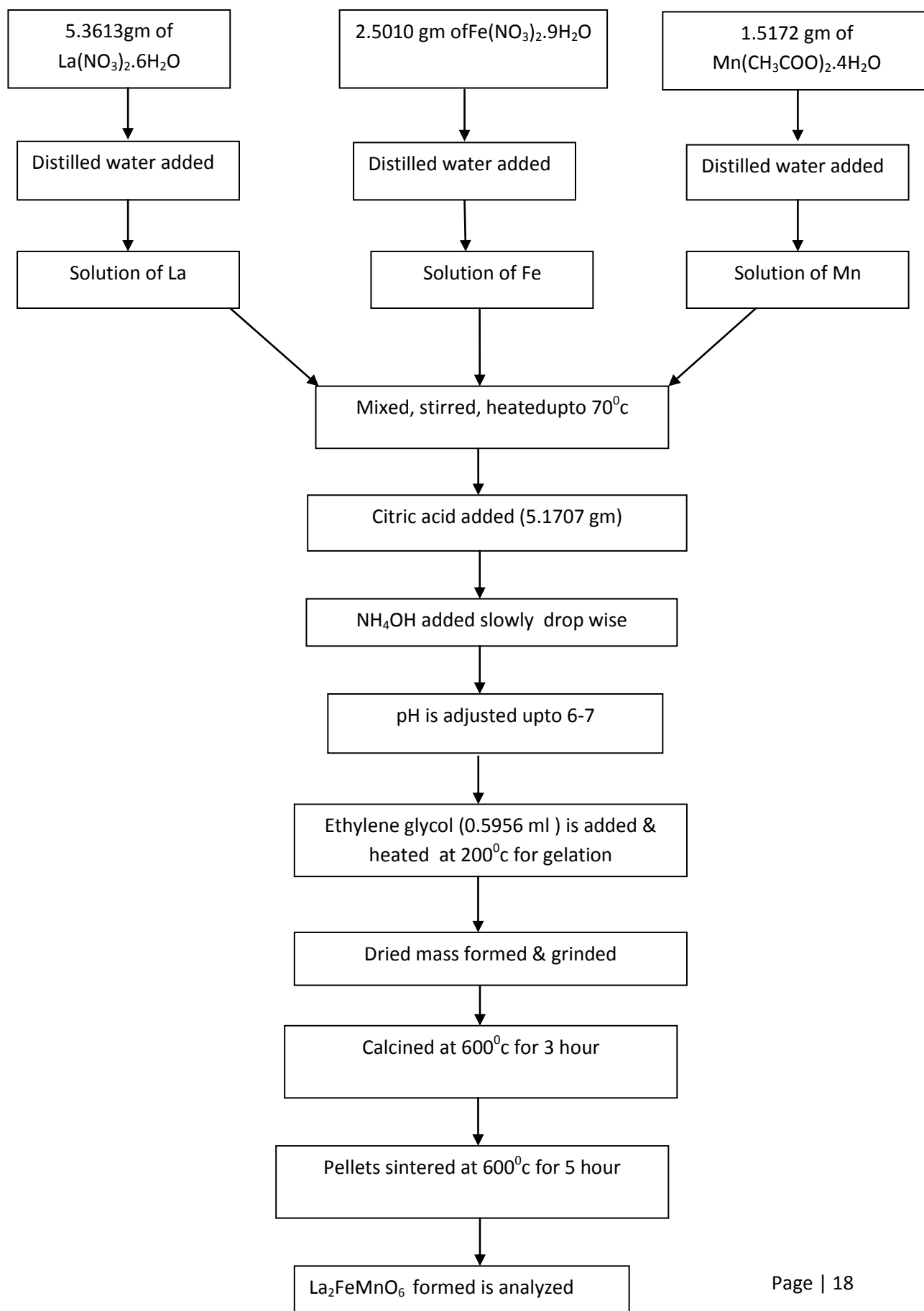
2.7.3 Synthesis of LaFeO_3 by sol-gel process:-



2.7.4 Synthesis of $\text{La}_2\text{CoMnO}_6$ by sol-gel route:-



2.7.5 Synthesis of $\text{La}_2\text{FeMnO}_6$ by sol-gel route:-



CHAPTER 3 SAMPLE CHARACTERIZATION

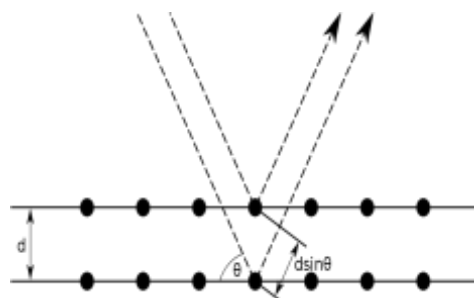
- X-Ray Diffraction technique
- UV-Visible Spectroscopy
- Field Emission Scanning Electron Microscopy
- Dielectric measurement
- FTIR

3.1 X-Ray diffraction technique

The properties of a material can be linked by the arrangement of atoms in its crystal structure. X-ray diffraction is a non-destructive analytical technique which can yield the unique fingerprint of Bragg reflections associated with a crystal structure.

One can regard a crystal structure as being built of layers, or planes, where each act as a semi-transparent mirror. X-rays with a wavelength similar to the distances between these planes can be reflected such that the angle of reflection is equal to the angle of incidence. We call this behaviour 'diffraction', and it is described by Bragg's Law.

When X-rays are incident on an atom, they make the electronic cloud move as does any electromagnetic wave. The movement of these charges re-radiates waves with the same frequency; this phenomenon is known as Rayleigh scattering. The re-emitted wave fields interfere with each other either constructively or destructively, producing a diffraction pattern on a detector or film. The resulting wave interference pattern is the basis of diffraction analysis. This analysis method is called Bragg diffraction.



According to Bragg the incident X-ray radiation would produce a Bragg peak if their reflections off the various planes interfered constructively. The interference is constructive when the phase shift is a multiple of 2π ; this condition can be expressed by Bragg's law.

$$2d \sin\theta = n\lambda$$

where n is an integer,

λ is the wavelength of incident wave

d is the spacing between the planes in the atomic lattice,

θ is the angle between the incident ray and the scattering planes.

3.2 UV visible spectroscopy

It is a absorption spectroscopy or reflectance spectroscopy in the ultraviolet-visible spectral region. This means it uses light in the visible and adjacent ranges. The colours of the chemicals involved are affected by the absorption or reflectance in the visible range. Molecules are undergoing electronic transitions in this region of electromagnetic radiation. In this technique, absorption measures transitions from the ground state to the excited state.

The basic principle of UV absorption is the molecule absorbs energy in the form of electromagnetic radiation to excite the electron to higher anti bonding molecular orbitals .the more easily the electrons are excited the longer the light can be absorbed. Different molecules absorb radiation of different wavelengths. An absorption spectrum will show a number of absorption bands corresponding to structural groups within the molecule. Its an absorption spectroscopy form 160-780 nm.

Beer–Lambert law is most often used in a quantitative way to determine concentrations of an absorbing species in solution, according to this law,

$$A = \log_{10} (I_0/I) = \epsilon CL$$

where A- measured absorbance

I_0 -intensity of incident light

I-intensity of transmitted light

L-path length through the sample

C -concentration of the solution

The instrument used in ultraviolet-visible spectroscopy is called as UV/Vis spectrophotometer. It measures the intensity of light passing through a sample (I), and compares it to the intensity of light before it passes through the sample (I_0). The ratio I/I_0 is called the transmittance, and is usually expressed as a percentage (%T). The absorbance, A , is based on the transmittance T , which is shown below,

$$A = -\log_{10}(\%T/100\%)$$

The UV-visible spectrophotometer can also be configured to measure reflectance. In this case, the spectrophotometer measures the intensity of light reflected from a sample (I), and compares it to the intensity of light reflected from a reference material (I_0). The ratio is called the reflectance, and is usually expressed as a percentage (%R).

3.3 FESEM & EDAX

The field emission scanning electron microscope is a versatile, non-destructive technique that gives detailed information about the morphology and composition of materials. FESEM produces clearer, less electrostatically distorted image with spatial resolution up to nano meter range. We can also get information about semiconductor device cross section analyses for gate widths, gate oxides, and film thicknesses.

FUNCTION

First the samples are gold pasted for the performance of FESEM. Electrons are ejected in a high electric field gradient from a field emission source. These electrons are also called as primary electrons. These primary electrons are focused and deflected by electronic lenses to produced narrow beam that bombarded on the sample. Due to this bombardment secondary electrons are ejected out from the sample surfaces. The angle and velocity of secondary electrons are related to the surface structure of the sample. A detector detects these secondary electrons and produces an electrical signal. This signal is amplified and transformed to a video scan image that can be seen on the screen and the digital image can be saved.

EDAX

This is an analytical technique used for the elemental analysis or compositional analysis of a sample. Here a high energetic electron is bombarded on the sample. Due to this interaction an inner shell electron is ejected out. To fill the vacancy electron having higher energy will jump down by radiating x-rays. All the elements have different wavelength x-rays. So by detecting these x-rays by a x-ray detector, we can detect the elements present in the sample.

3.4 FTIR

Fourier transform infrared spectroscopy (FTIR) is a technique which is used to obtain an infrared spectrum of absorption, emission, photoconductivity or Raman scattering of a solid, liquid or gas. An FTIR spectrometer simultaneously collects spectral data in a wide spectral range. It is so named because Fourier transform is required to convert the raw data into actual spectrum. To obtain the data it uses an interferometer.

A beam generated by the broad band light source shines into the Michelson interferometer with certain configuration of mirrors. One of which is moved by a motor. When the mirror moves the interferometer blocks, transmits each wavelength of light present in the beam periodically due to wave interference. Differently modulated wavelengths are the cause of producing the beam with different spectrum. The data of the interferometer “interferogram” is modified by using Fourier transform. The Fourier transform defines a relationship between signal in time domain & its representation in frequency domain. Being a transform of a signal is a continuous complex valued signal capable of representing real valued continuous time signal.

3.5 DIELECTRIC SPECTROSCOPY:

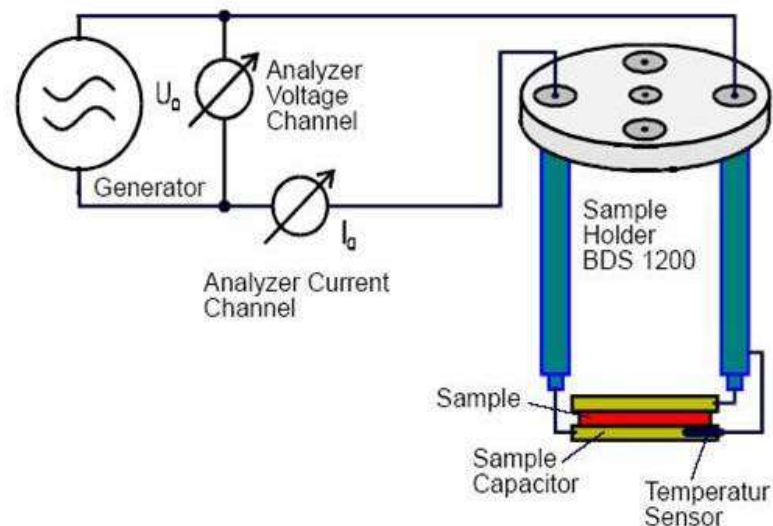
Impedance spectroscopy also known as Electrochemical Impedance Spectroscopy (EIS), measures the dielectric properties of a medium as a function of frequency. It is based on the interaction of an external field with the electric dipole moment of the sample, often expressed by permittivity. Dielectric constant is defined as to be the measure of the polarisability of a material that is how strongly it resists the movement of polar molecule. Permittivity can be expressed as a complex quantity with a real and an imaginary part.

$$\varepsilon = \varepsilon' - \varepsilon''$$

Tangent loss is given by

$$\tan\delta = \varepsilon'' / \varepsilon'$$

Impedance measurements are a basic means of evaluating electronic components and materials. Every material has a unique set of electrical characteristics that are dependent on its dielectric or insulation properties. The dielectric property analysis was conducted by using an Impedance/Gain Phase Analyser.



By this method we are measuring the capacitance (C), dielectric loss (D), impedance (Z) of the sample. Using these data we are calculating the dielectric constant of the sample from the equation. Given below

$$C = (\varepsilon_0 \varepsilon_r A) / d$$

Where ε_0 is the permittivity in free space

ε_r is the dielectric constant

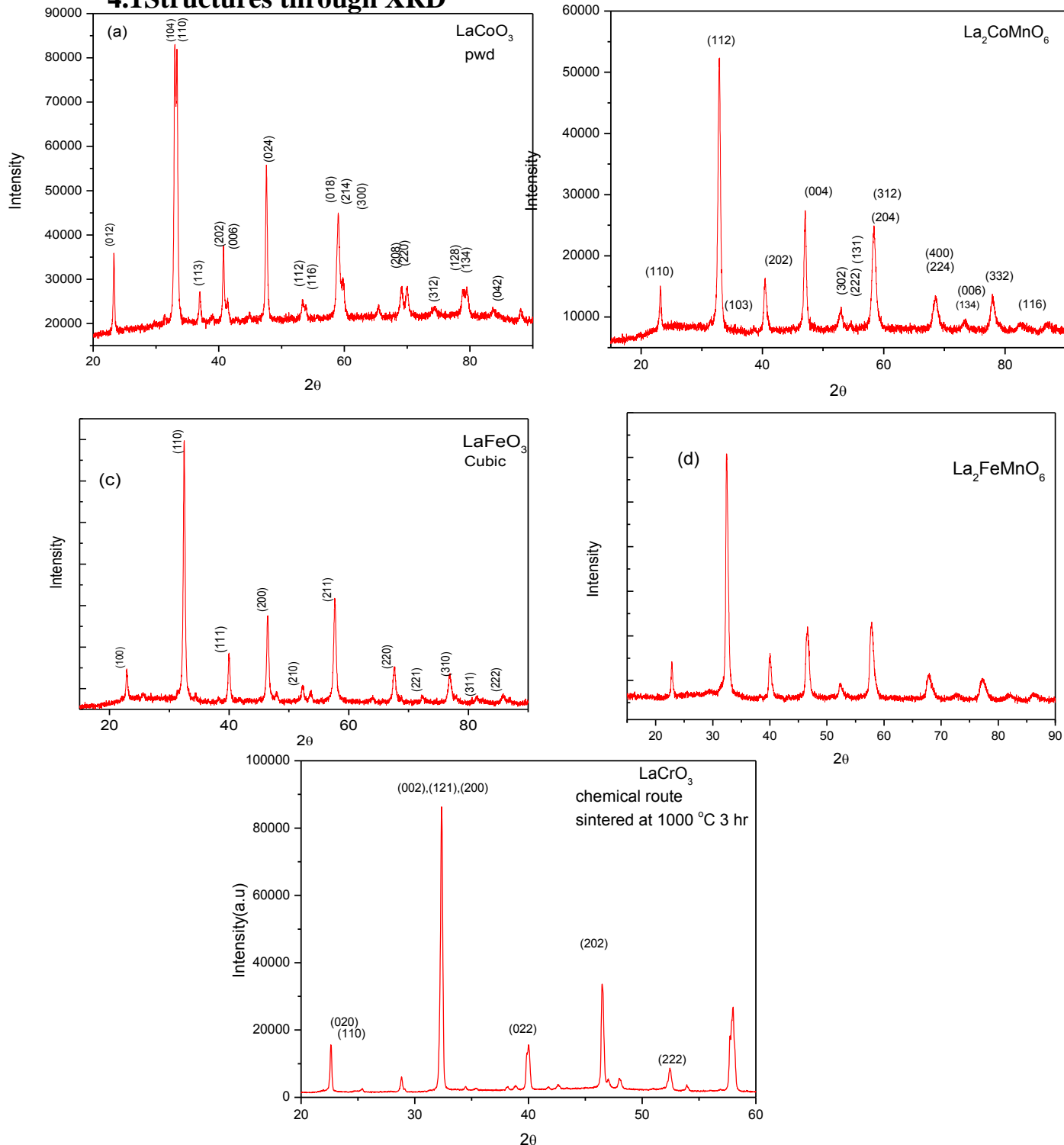
A is the surface area of the sample

D is the thickness of the sample

CHAPTER-4

RESULTS AND DISCUSSIONS

4.1 Structures through XRD



From the XRD data analysis, the structure of LaCoO_3 , LaCrO_3 , LaFeO_3 is found to be rhombohedral, orthorhombic, cubic respectively.

4.2 Bandgap information from UV-Visible spectra

From the UV-visible data it is confirmed that the double perovskite compounds are semiconducting in nature due to its band gap is found below 2 eV. And for the perovskite s it is found to be above 3 eV, which indicates their insulating behaviour.

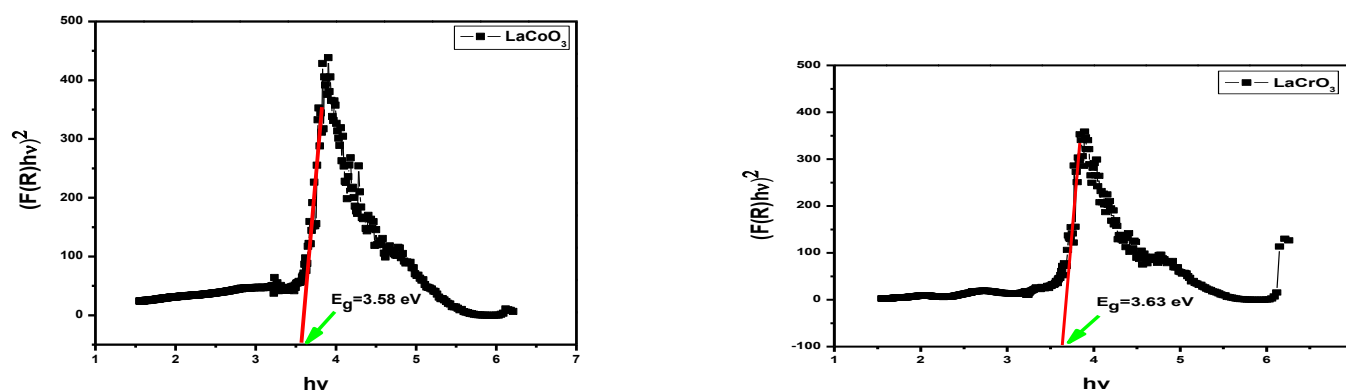


Fig. 4.3.1 UV-visible graph for $\text{LaCoO}_3, \text{LaCrO}_3$

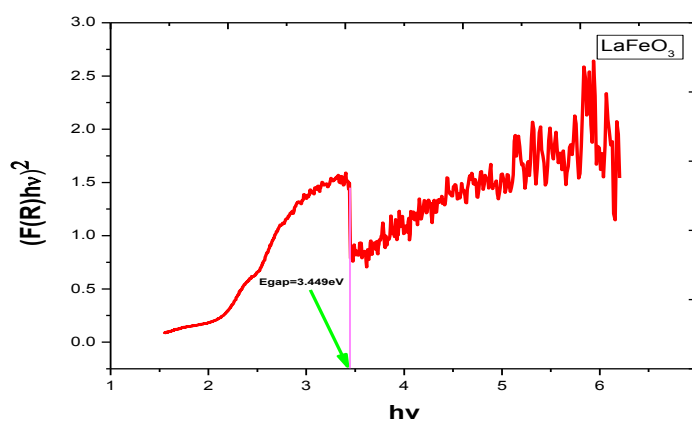


Fig. 4.3.2 UV-visible graph for LaFeO_3

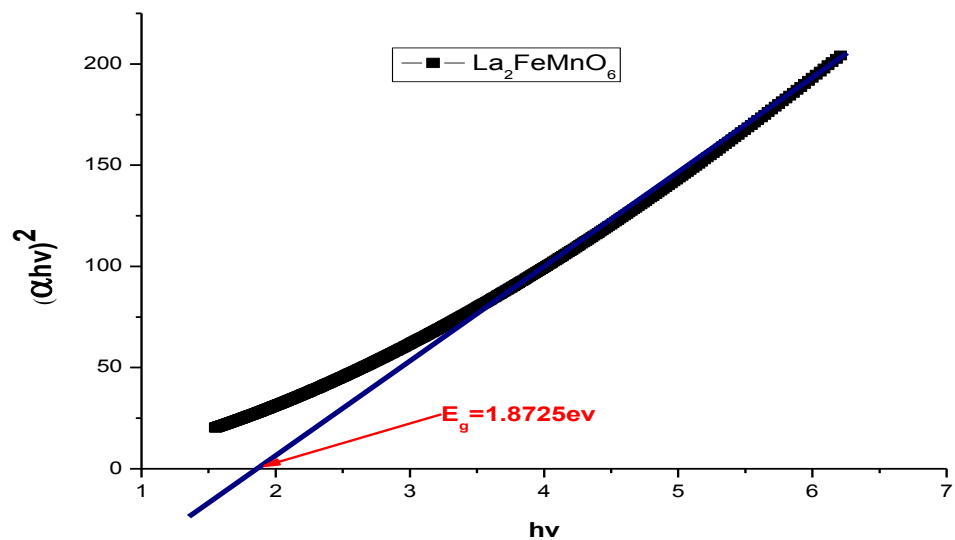


Fig. 4.3.3 UV-visible graph for $\text{La}_2\text{FeMnO}_6$

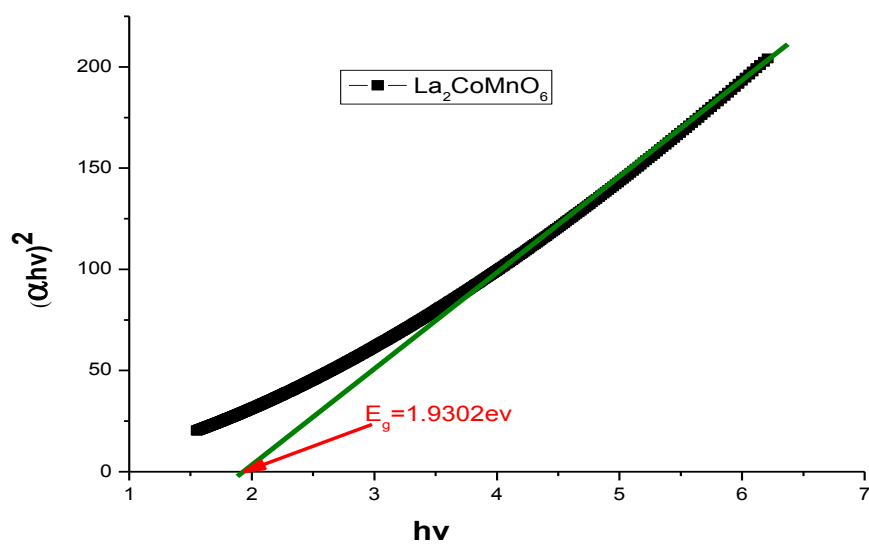


Fig. 4.3.1 UV-visible graph for $\text{La}_2\text{CoMnO}_6$

4.3 Microstructures from FESEM

The morphology of the double perovskite compounds has been studied through FESEM. From that the size of the grains is found to be in micron. In addition there is porosity found in samples. This is probably due to the low sintering temperature of the materials.

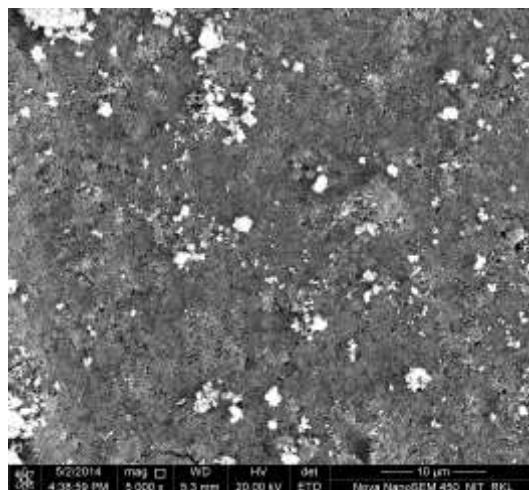
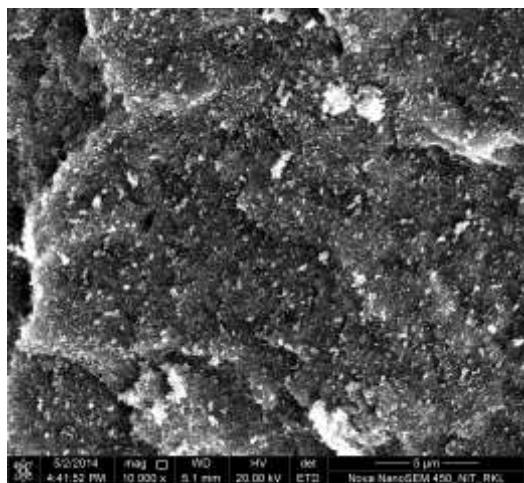
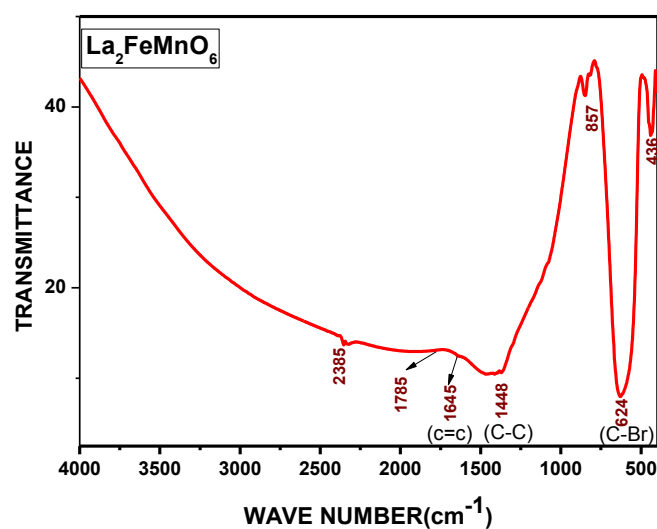
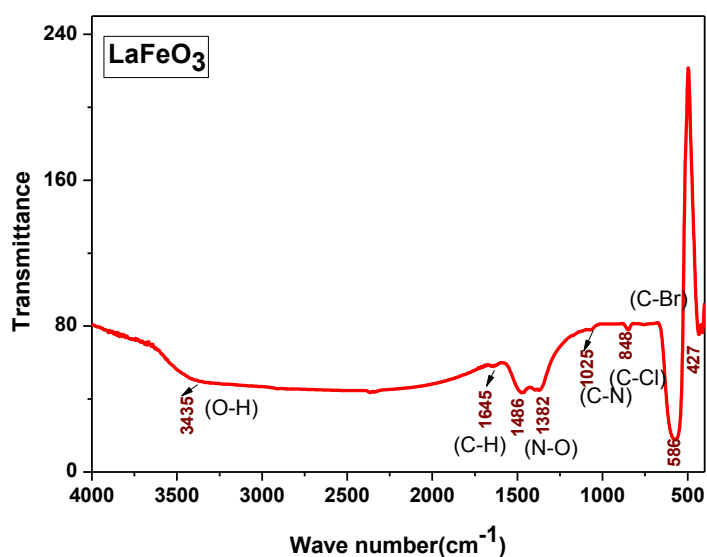
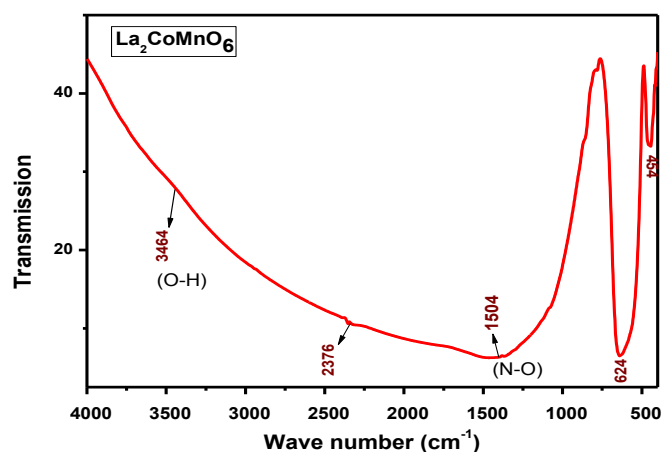
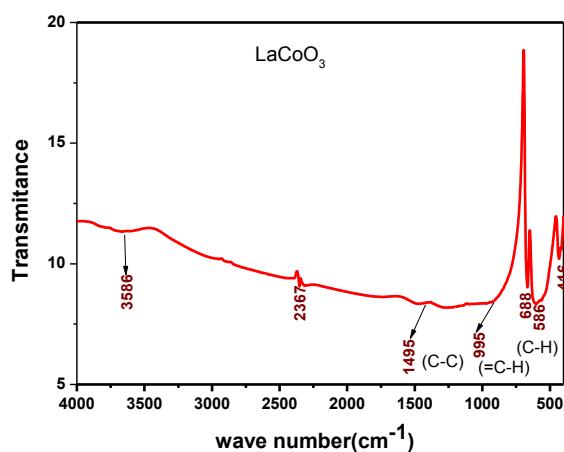


Fig 4.3.1 FESEM of La₂CoMnO₆ Fig. 4.3.2 FESEM of La₂FeMnO₆

Moreover, the EDAX spectra confirm the presence of elements in the respective compounds which is tally with their atomic weight percentage.

4.4 Bond nature by FTIR



From the above FTIR data we knew about the different bonding in our samples. In LaFeO_3 the bonds C=C are formed in the range $1400\text{--}1600\text{cm}^{-1}$, they are stretched in the weak intensity. In $\text{La}_2\text{FeMnO}_6$ the bonds C=C are formed in the range $1400\text{--}1600\text{cm}^{-1}$ with strong intensity.

4.4 Dielectric spectroscopy

The material capacitance (source of dielectric constant) and loss has been plotted as a function of frequency for both LaFeO_3 and $\text{La}_2\text{FeMnO}_6$ at room temperature. With increase in frequency the dielectric constant decreases as similar to many of the dielectric materials.

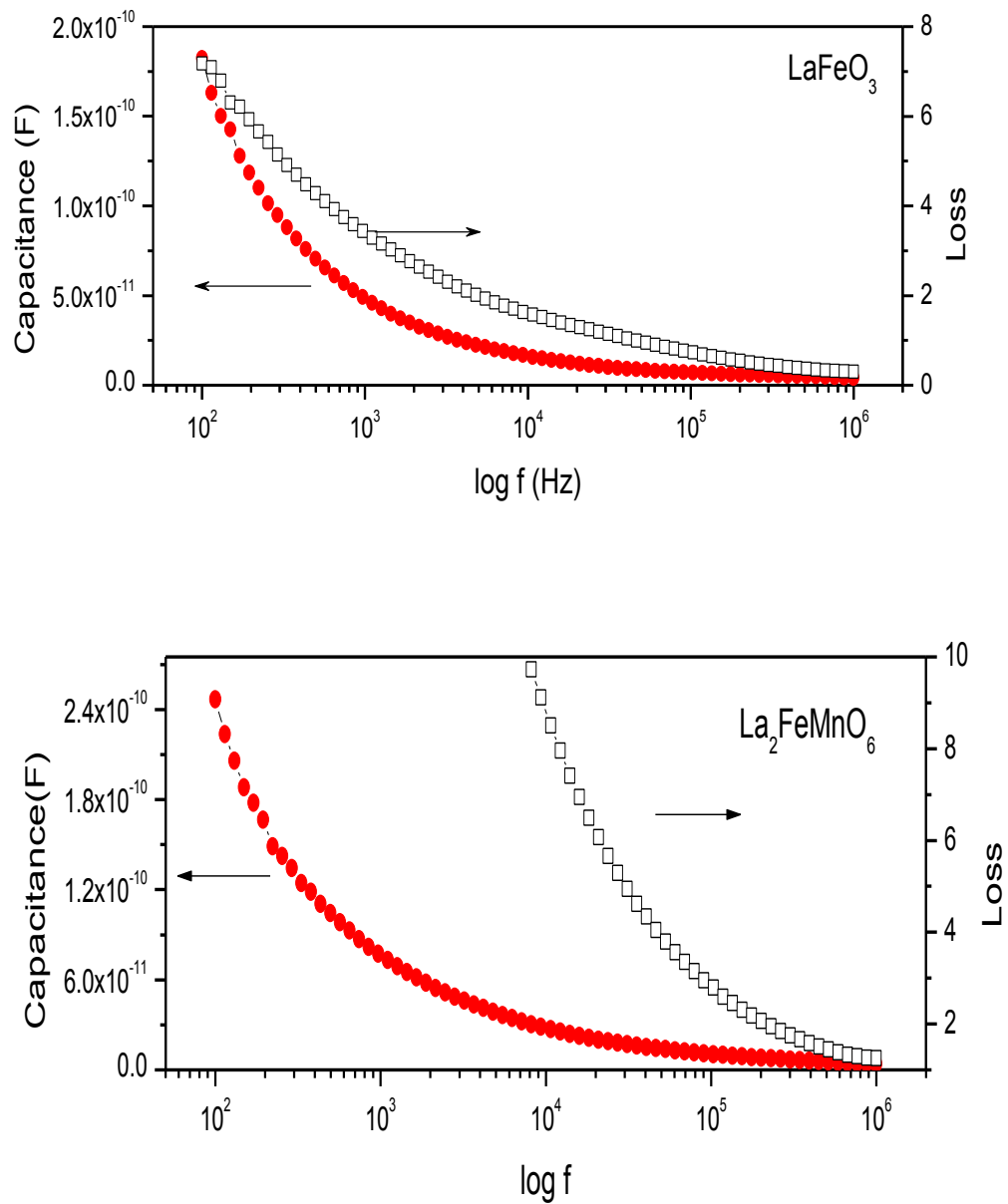


Fig 4.4.1: Capacitance and loss tangent vs log of frequency for $\text{La}_2\text{FeMnO}_6$ and LaFeO_3

Capacitance & loss factor vs. Log f plot also drawn for both LaCoO_3 and $\text{La}_2\text{CoMnO}_6$. And it was found that both are decreasing with increase in frequency.

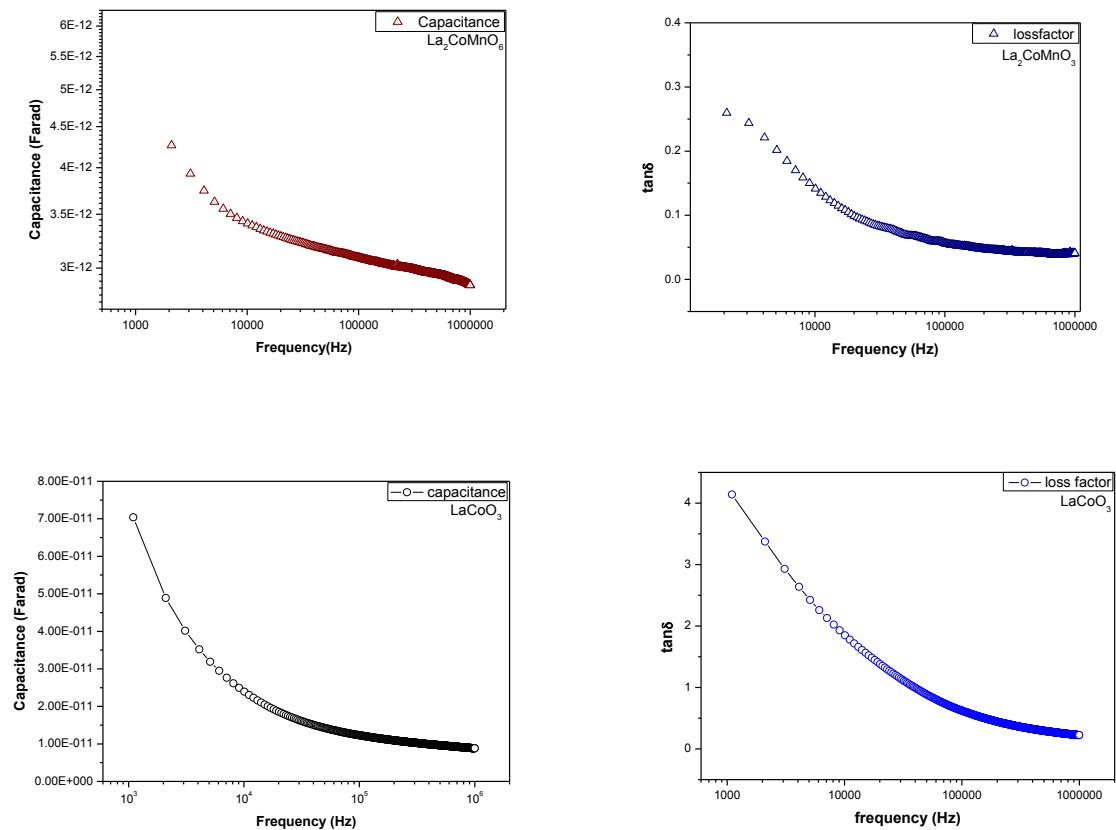


Fig 4.4.2: Capacitance and loss tangent vs log of frequency for $\text{La}_2\text{CoMnO}_6$ and LaCoO_3

CHAPTER-5 CONCLUSION AND SCOPE OF FUTURE WORK

We have studied the structural and electrical properties of several perovskite and double perovskite compound and also got how properties changes from perovskite compound to double perovskite compound significantly. Compounds are synthesized by sol-gel process and pyrophoric process. The samples are found to be in single phase and stoichiometric. The information about the molecular bonding is obtained from the FTIR spectra. The band gap is calculated from UV-Visible spectra characterises the semiconducting and insulating nature of the material. Perovskite compound behaves like insulator, when manganese is doped with these compounds it behaves like semiconductor. The morphology of the materials has been studied through FESEM and finally the dielectric spectroscopy has been carried out for all compounds.

Furthermore, one can study the correlation between different electric and magnetic order parameter for this materials, to use as a potential candidate for multiferroics. The magnetization dynamics can be studied to know the spin-spin interactions. Low temperature dielectric spectroscopy can be studied to know whether this materials undergoes any transition or not and if so what is the inside story of this transition and many more work can be done related to these materials.

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